Tab 1

How to Etch an STM Tip

Lab Manual

By: Chance LaVoie

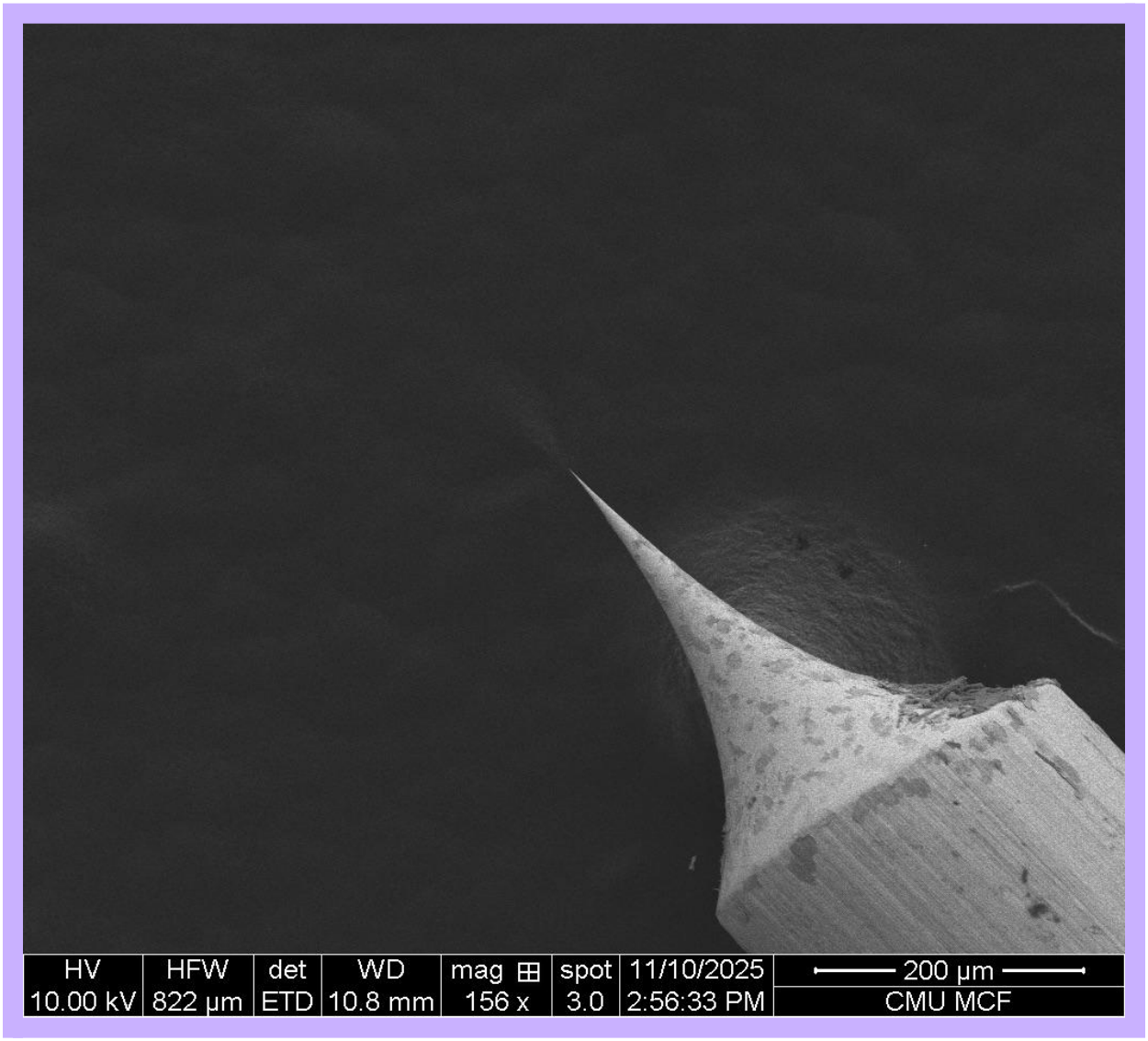


Table of Contents:

Safety Notes

Required Personal Protective Equipment (PPE)

All etching work must be performed while wearing full laboratory PPE. This includes: **a lab coat**, **long sleeves, long pants**, **closed-toe shoes, safety glasses, and gloves**. PPE must remain on at all times while preparing solutions, handling reagents, or operating the etching setup.

Chemical Hazards

This procedure requires handling solid sodium hydroxide (NaOH) pellets, 2 M aqueous NaOH**,** and the reaction products formed during the alkaline etching of tungsten. Sodium hydroxide—both in pellet form and in solution—is **strongly corrosive**. Contact with skin or eyes can result in **severe chemical burns,** and inhalation of aerosols or vapors may cause **respiratory irritation**. Exercise caution when transferring pellets, preparing solutions, and disposing of spent etchant.

Safety Data Sheets (SDS)

Before beginning the procedure, review the current Safety Data Sheets (SDS) for all relevant substances and ensure that they are accessible throughout the experiment.

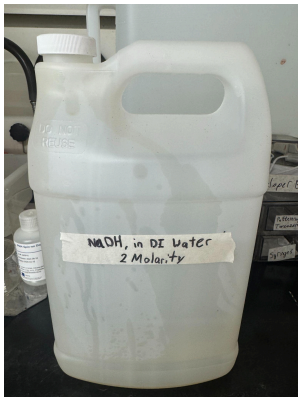
- Sodium hydroxide pellets: *SDS Link*

- 2 M sodium hydroxide solution: *SDS of a comparable 2M NaOH solution*
- Note about Etching biproducts:
    - Etching tungsten in sodium hydroxide generates dissolved tungsten species—primarily sodium tungstate—along with minor amounts of tungsten oxides, suspended particulate residue, and hydrogen gas. The spent etchant remains strongly alkaline and must be handled and disposed of as hazardous chemical waste in accordance with institutional protocols.

Waste Disposal

Aqueous Waste (Spent Etchant)

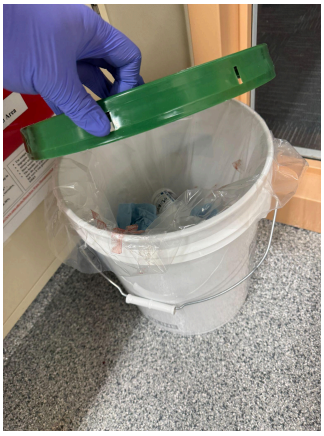
Following the etching process, all liquid waste—including the used 2 M NaOH solution, dissolved tungsten species and any suspended particulates—must be transferred into the designated, labeled **liquid waste container.** The waste bottle must remain in its **secondary containment tray inside the fume hood** at all times.



Never pour down the drain! Spent etchant must be managed as hazardous chemical waste according to institutional EHS procedures.

Solid Waste

Any solids contaminated with NaOH or etch products—such as gloves, wipes, paper towels, broken tungsten tips, or filter residues—must be disposed of in the laboratory's **solid hazardous-waste bucket** (green-lidded container).
Ensure the lid is fully secured after use and that no liquids are placed in this container.



Ventilation and Engineering Controls

All steps involving NaOH preparation and tungsten etching must be conducted in a **fume hood**. The etching reaction can release gases and caustic aerosols; using a fume hood prevents inhalation exposure and avoids the accumulation of hazardous vapors in the laboratory environment.

Equipment and Materials

The following equipment and materials are required to prepare the etchant, set up the apparatus, operate the etching system, and safely collect waste.

Personal Protective Equipment (PPE)

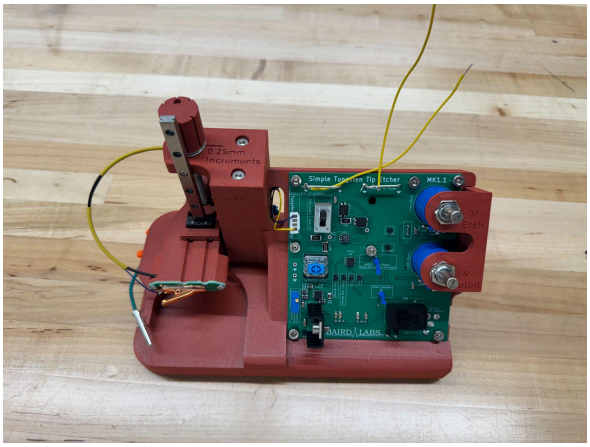
(Required throughout all steps of preparation, etching, and cleanup).

- Lab coat
- Long sleeves and long pants
- Closed-toe shoes
- Safety glasses
- Gloves

Etching Apparatus and Electronics

(Components used to assemble and operate the tip-etching system)

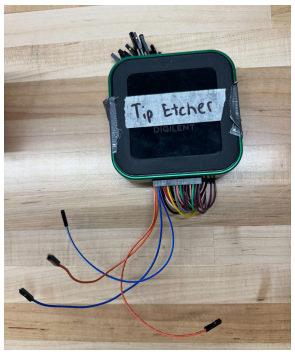
- Tip etcher assembly



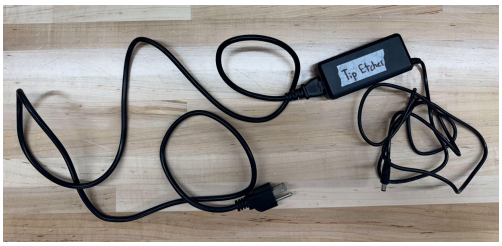
- Glass beaker with cathode ring

- USB-C cable

- Analog Discovery 3



- 18 V power supply



- Computer with the working Python GUI

Chemicals and Solution Preparation Tools

(Required for preparing the 2 M NaOH etching solution)

- Sodium hydroxide (NaOH) pellets

- Deionized (DI) water

- Beaker for mixing NaOH solution

- Graduated cylinder for measuring DI water

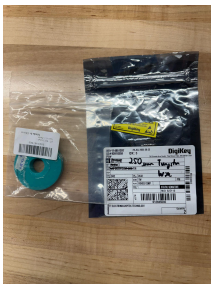
- Electric scale (for weighing NaOH pellets)

- Delicate task wipes

Materials for Etching and Handling

(Physical components used during tungsten-wire mounting and etching)

- Tungsten wire



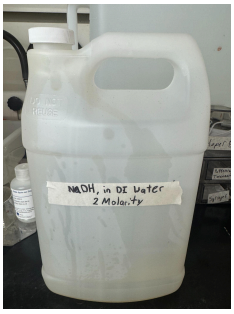
- Tweezers (for positioning and retrieving wire pieces)

- Wire Cutters (for cutting the Tungsten wire)

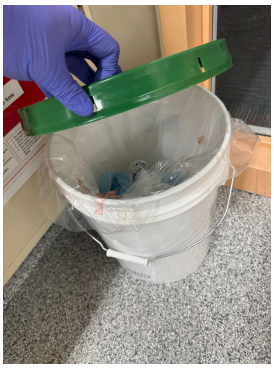
Waste Collection Containers

(Designated containers used during and after etching)

- Liquid waste container for spent NaOH/tungstate



- Solid hazardous-waste container (green-lidded bucket)



GUI Setup

Download WaveForms

Go to the Digilent WaveForms download page:
https://cloud.digilent.com/myproducts/waveforms?pc=1&tab=2

→ Make an account if required, or login.

Download the WaveForms installer for your operating system.

Install WaveForms (macOS Example)

Open the downloaded WaveForms.dmg file.

In the installation window:

1. Drag WaveForms into the Applications folder.
   (This installs the GUI.)
2. Drag dwf.framework into your Frameworks folder.
   a. (This installs the runtime that allows WaveForms to talk to the Analog Discovery 3.)

Download the Python Control Script and Requirements File

The automated etching interface is provided as a standalone Python script. You must download this script before configuring the software environment.

1. Navigate to the project GitHub repository: https://github.com/cmuchancel/HackerFabTipEtcher
2. In the repository, download the files named:
    - `HackerFabSTMTipEtch.py`
    - `requirements.txt`
3. Create a new folder in a convenient and permanent location on your computer (for example: *Documents/STM_Etcher/*). This folder will hold:
    i. the Python control script
    ii. requirements file
    iii. log files/plots
    iv. your virtual environment (created next)

Setting Up the Python Virtual Environment

A Python virtual environment ensures that all dependencies required for the etching GUI remain isolated from your system installation.

Create the Virtual Environment

Open a terminal and navigate to the project folder:

```
cd /path/to/your/project/folder
```

Create a virtual environment named venv:

```
python3 -m venv venv
```

Activate the Virtual Environment

macOS / Linux:

```
source venv/bin/activate
```

Windows (PowerShell):

```
venv\Scripts\Activate.ps1
```

When activated, your prompt will show:

```
(venv)
```

Install Dependencies from requirements.txt

With the virtual environment active, install the required packages using:

```
pip install -r requirements.txt
```

This installs all modules needed by the GUI, including:

```
WF_SDK>=1.2.0
matplotlib>=3.5
```

Procedure

Setup & Power-On

Step 1 — Put on PPE

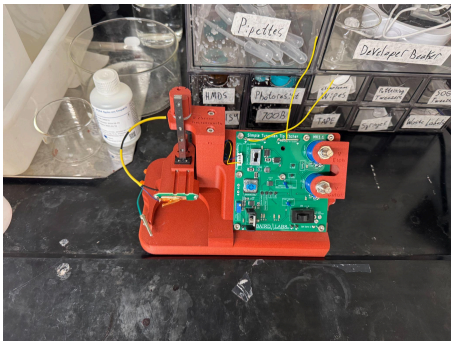
Put on full personal protective equipment (PPE): lab coat, long sleeves, long pants, closed-toe shoes, safety glasses, and chemical-resistant gloves.

Step 2 — Gather Equipment and Materials

Collect all required items from the Equipment and Materials section before beginning the setup.

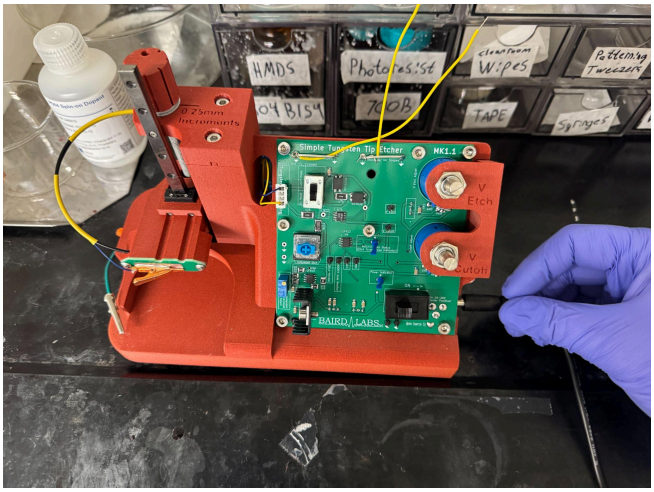
Step 3 — Place the Tip Etcher Assembly in the Fume Hood

Move the tip etcher assembly into the fume hood and position it on a stable, level surface.



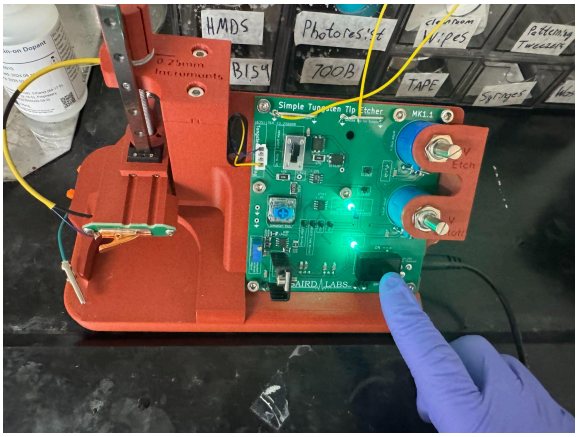
Step 4 — Connect the 18 V Power Supply to the Tip Etcher

Plug the 18 V power supply into the tip etcher.



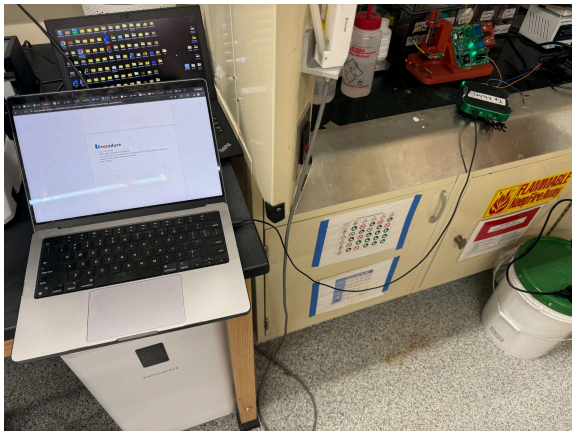
Step 5 — Turn the Tip Etcher ON

Flip the power switch on the tip etcher to the ON position. Green lights should turn on.



Step 6 — Connect the Analog Discovery 3 to the Computer

Using the USB-C cable, connect the Analog Discovery 3 to the computer that will run the Python GUI.



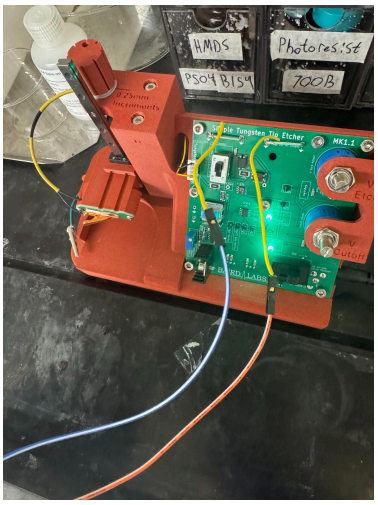
Step 7 — Connect Grounds (Channel 1– and Channel 2–)

Connect the negative terminals from the Analog Discovery 3:

Channel 1– (orange/white)

Channel 2– (blue/white)

to the yellow ground wires on the tip etcher assembly



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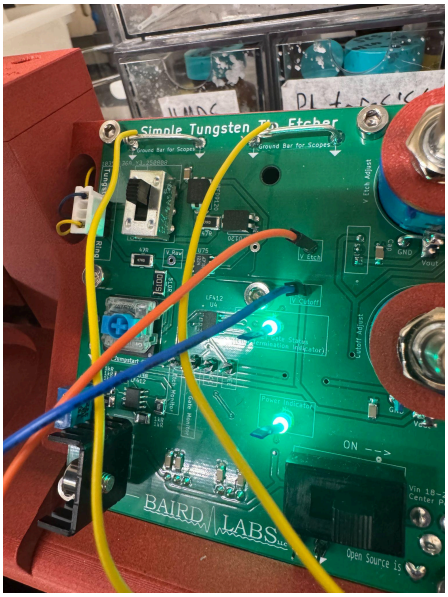
Step 8 — Connect Signal Outputs (Channel 1+ and Channel 2+)

Connect the positive terminals:

Channel 1+ (solid orange) → V_etch

Channel 2+ (solid blue) → V_cutoff

Verify that each wire is fully seated and secure.



Step 9 — Navigate to the Tip Etcher Directory

Open a terminal window and navigate to the directory containing your Python control script and requirements.txt file.

```
cd /path/to/your/TipEtcherDirectory
```

Step 10 — Activate the Virtual Environment

Activate your project's virtual environment:

macOS / Linux:
```
source venv/bin/activate
```

Windows (PowerShell):
```
venv\Scripts\Activate.ps1
```

Ensure the terminal prompt shows (venv).

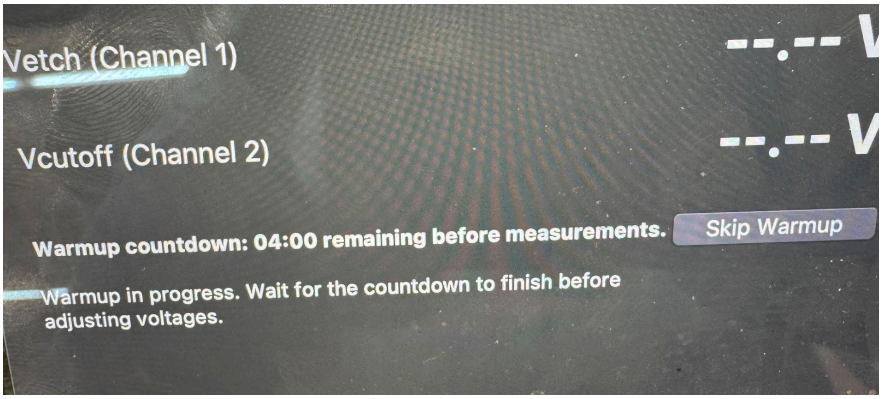
Step 11 — Launch the Python GUI

Run the tip-etching GUI:
```
python HackerFabSTMTipEtch.py
```

The GUI should open and automatically connect to the Analog Discovery 3.

Step 12 — Allow Electronics to Reach Thermal Equilibrium

Use the GUI's built-in timer to let the electronics stabilize for 5 minutes before beginning calibration or etching.



Prepare the Etching Solution

Step 13 — Prepare the Etching Solution

While the electronics warm up, begin preparing the NaOH etching solution following steps 14-16.

Step 14 — Measure 20 mL of DI Water

Using a graduated cylinder for accuracy, measure **20 mL of deionized (DI) water** and transfer it into a clean mixing beaker.

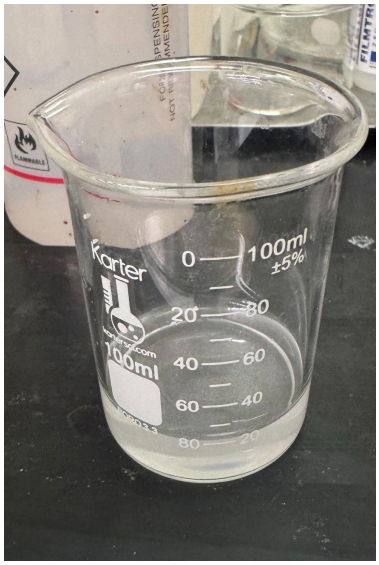
Step 15 — Weigh Out 1.6 g of NaOH Pellets

Using the electronic scale, **weigh 1.6 g of sodium hydroxide (NaOH) pellets.**
Place a laboratory wipe on the scale to act as a clean weighing surface, then tare the balance before adding pellets.

Step 16 — Dissolve the NaOH Pellets in the DI Water

Transfer the NaOH pellets into the beaker containing the DI water.
Gently swirl the beaker until the pellets are fully dissolved.

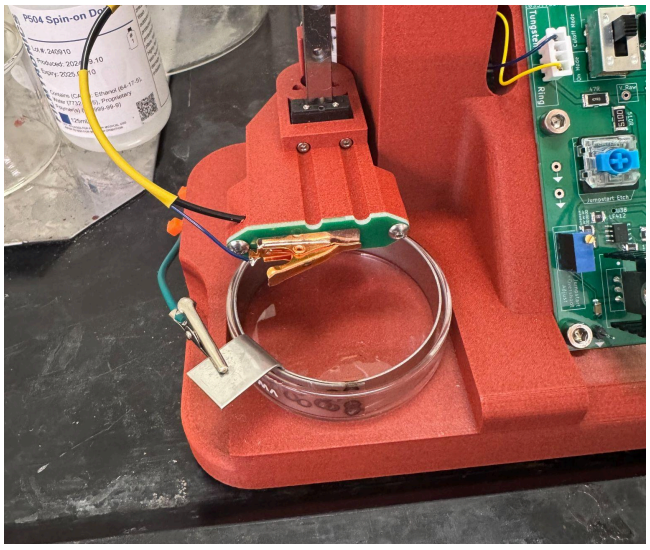




Prepare the Etching Assembly

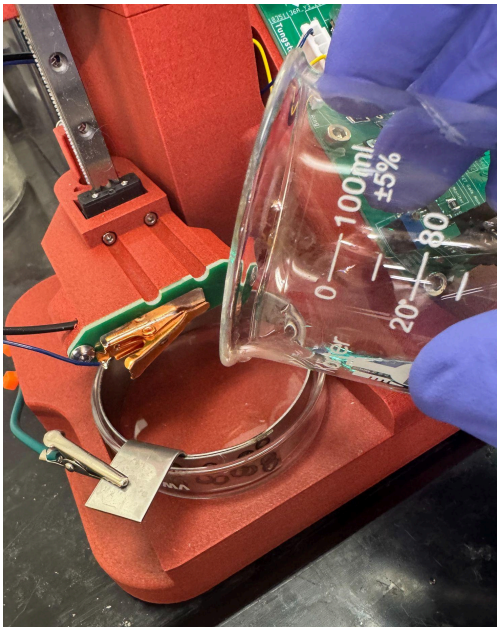
Step 17 — Set Up the Etching Beaker and Electrode Ring

Clean the Etching Beaker and Electrode ring thoroughly with DI water. Let it dry.
Place the etching beaker and electrode ring into the tip etcher assembly.
Attach the alligator clip from the etcher to the metal tab on the electrode ring to ensure proper electrical contact.



Step 18 — Add the Etching Solution to the Electrode Ring Beaker

Carefully pour the 20 mL NaOH solution into the etching beaker, ensuring that the electrode ring is fully submerged and that the solution level will cover the immersion depth of the tungsten wire during etching.



Step 19 — Cut the Tungsten Wire

Using wire cutters and tweezers, cut approximately 1 inch of tungsten wire. Handle the wire carefully to avoid bending or nicking it.

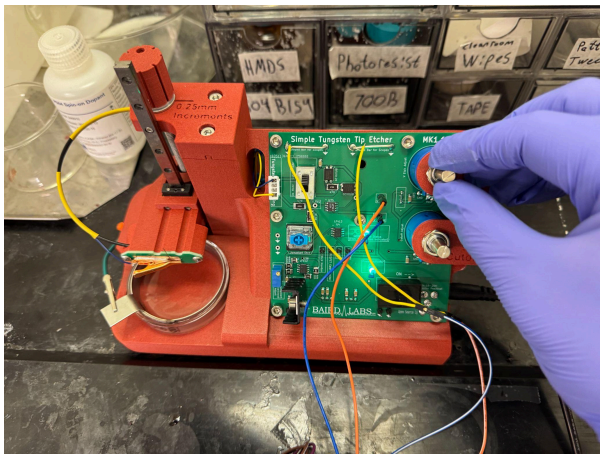
Step 20 — Mount the Tungsten Wire

Affix the tungsten wire to the clip on the left side of the tip etcher assembly.
Use tweezers to straighten the wire and ensure it hangs perfectly vertical and is centered above the etching beaker.

Pre-Etch Stability & Monitoring Setup

Step 21 — Tune V_etch and V_cutoff

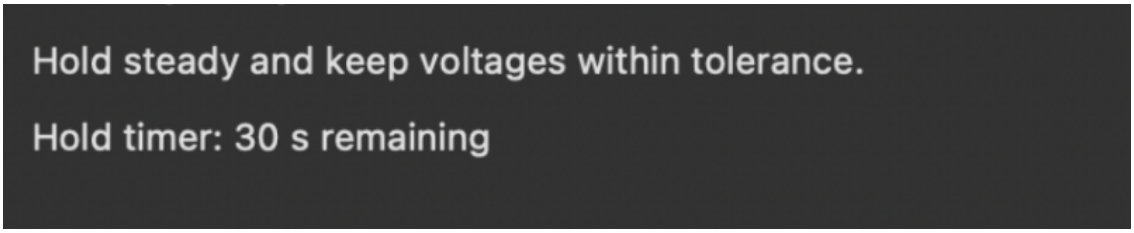
Using the adjustment dials on the tip etcher assembly, tune the voltages until the live readings on the GUI fall within tolerance of the desired setpoints(V_etch: 12 V , V_cutoff: 1.5V)
Make small adjustments to avoid overshooting.



Step 22 — Wait for the Voltage Hold Timer

Once both voltages are tuned and within tolerance, the GUI will automatically begin a 30-second hold timer.

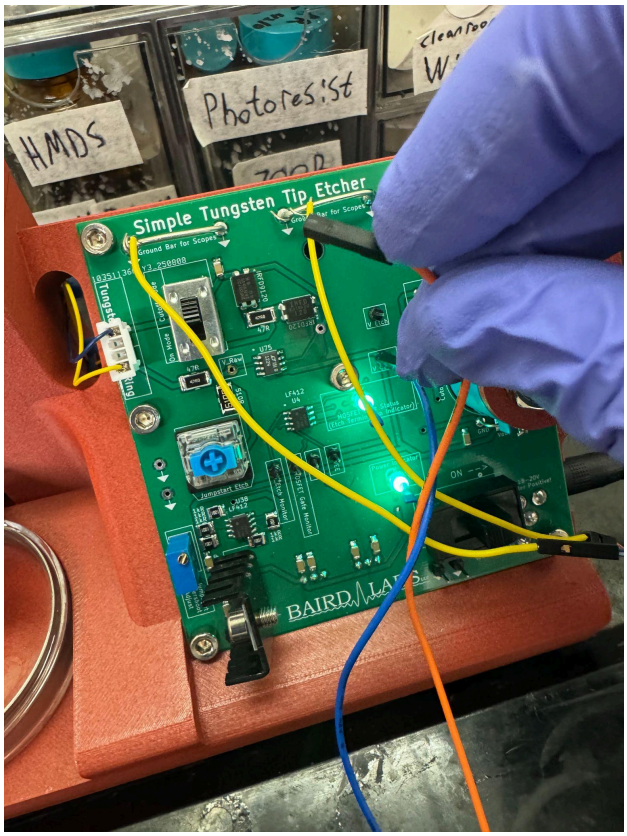
This ensures that the voltages remain stable before etching begins.

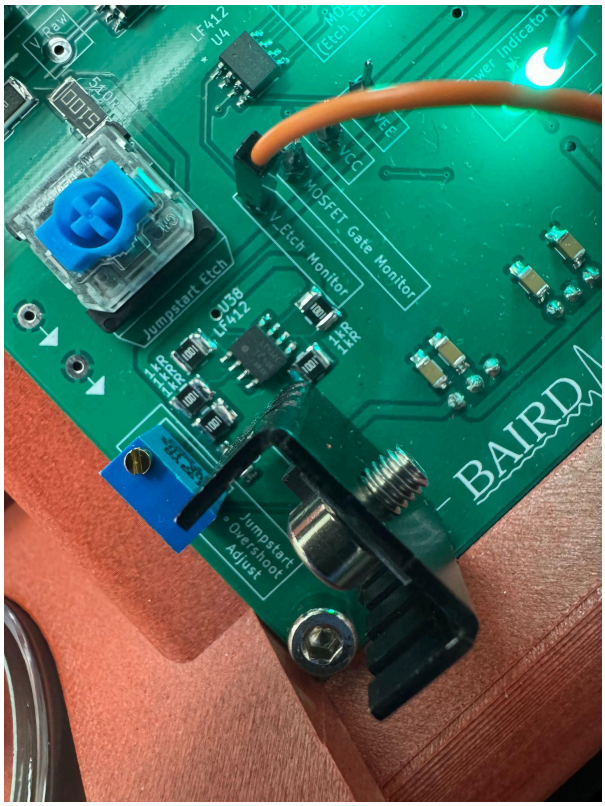


Step 23 — Move Channel 1+ to the Monitoring Input

To avoid contaminating the etch current, disconnect Channel 1+ (solid orange) from V_etch and reconnect it to V_etch Monitor.

Press the confirmation button in the GUI to verify the connection.

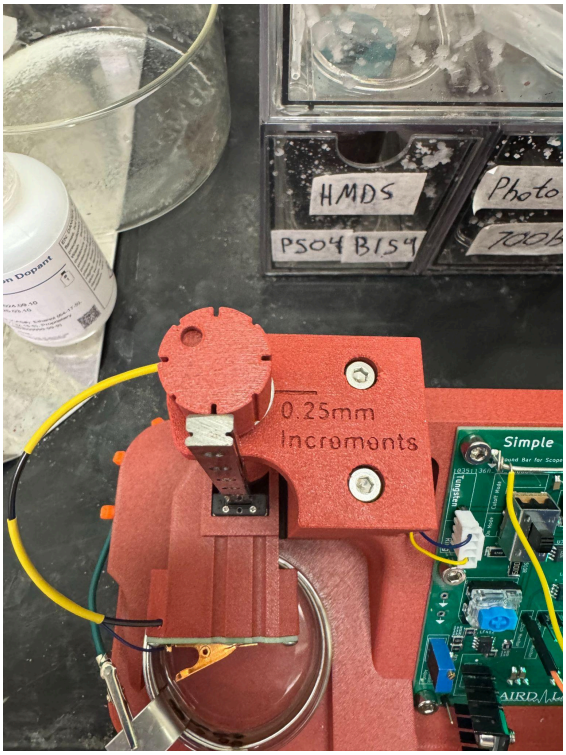


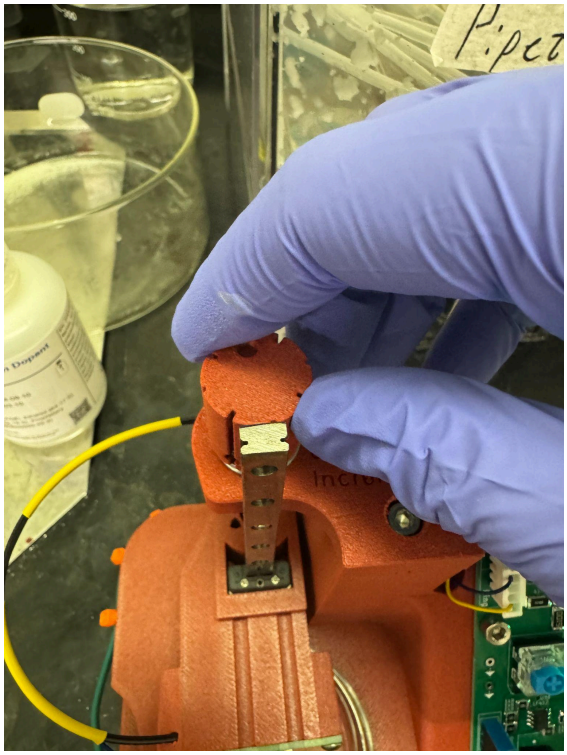


Step 24 — Lower the Tungsten Tip into the Etching Solution

Using the height-adjustment dial on top of the etcher:

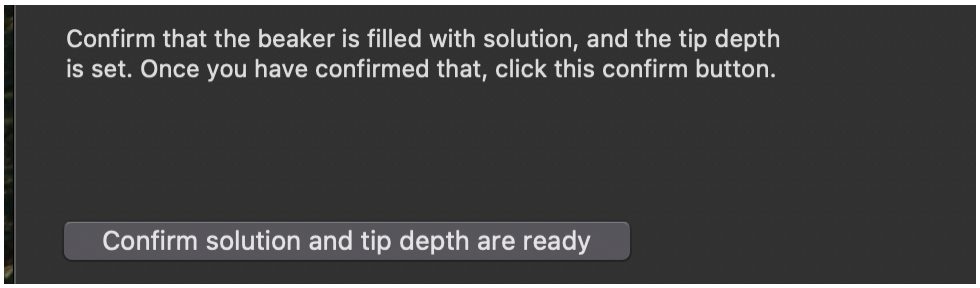
1. Rotate the knob counter-clockwise (CCW) until the tungsten tip just touches the surface of the NaOH solution and forms a visible meniscus.
2. Note the rotational position.
3. Rotate the knob six (6) additional detents (each 0.25 mm) CCW.
4. Final immersion depth: 1.5 mm below the surface.





Step 25 — Confirm Progress in the GUI

Click the confirmation button in the GUI indicating that the tip depth has been set and solution is ready.



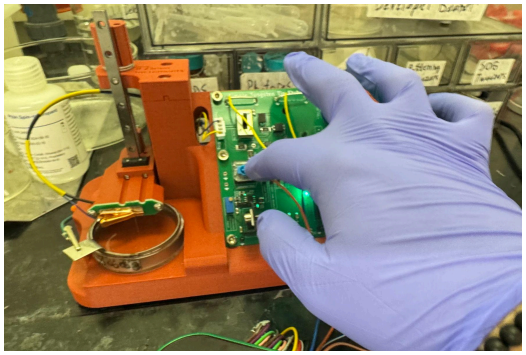
Etching Process

Step 26 — Begin the Etching Process

Press the blue button on the tip etcher to begin the etch.

- Avoid loud noises, footsteps, or any vibrations during this time—mechanical disturbances can degrade tip quality.

Once etching begins, the MOSFET gate status indicator should turn off.



Step 27 — Allow the Etch to Run to Completion

The Python GUI will automatically detect the start of the etch and begin monitoring the process.

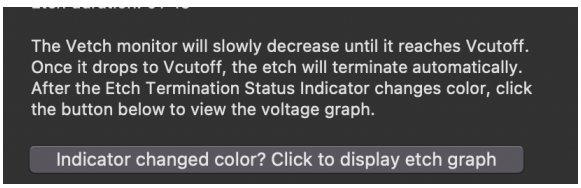
- Typical etching time: ~10 minutes
- You should observe the Vetch Monitor voltage gradually decreasing on the GUI
- When it drops below V_cutoff, the etcher will automatically terminate the process.

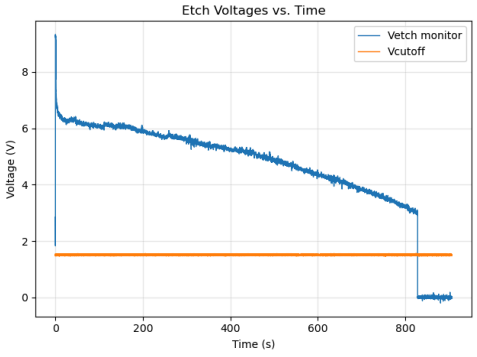
When etching ends, the MOSFET gate status light will turn green again.

Step 28 — Display and Save the Etch Graph

Click the GUI button confirming that the indicator has changed color to display the etching graph.

A matplotlib window will appear; you may save the graph from this viewer for documentation or further analysis.





Post-Etch Handling

Step 29 — Remove the Finished Tip

Turn the big dial CW to raise the tip out of the etchant.

Use tweezers to carefully remove the etched tungsten tip from the assembly.
Avoid touching the etched region.

Step 30 — Dispose of the Etching Solution and Clean the Apparatus

After removing the tungsten tip, carefully pour the used NaOH/tungstate etching solution into the designated liquid waste bottle kept in secondary containment inside the fume hood.
Never pour this solution down the drain!

Once the beaker is empty:

- Rinse both the electrode ring and the etching beaker thoroughly with DI water.
- Ensure all residue is removed and the components are visibly clean.
- Set the components aside and let them drip dry before storing or preparing for the next etch.

This cleaning step prevents contamination and ensures consistent etching results.

Step 31 — Turn off the Tip Etcher and Return all Equipment to Storage

Switch off the tip etcher and unplug the power supply. Disconnect all cables and return the equipment, tools, and materials to their designated storage locations. Ensure the workspace and fume hood are left clean and orderly.

Step 32 — Inspect the Etched Tip

Examine the tip under a microscope.
The etch graph may reveal subtle electrical disturbances or mechanical vibrations that occurred during the process, which can help diagnose issues or improve future etches.

Etching Procedure Complete!